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DETERMINATION OF NICKEL, NICKEL CHLORIDE HEXAHYDRATE, AND BORIC ACID IN NICKEL SULFAMATE PLATING SOLUTIONS BY TITRATION

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Determination Of Nickel, Nickel Chloride Hexahydrate, And Boric Acid In Nickel Sulfamate Plating Solutions By Titration

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Abstract

The chemical literature lacks analysis methods for the determination and adequate monitoring of nickel, nickel chloride hexahydrate and boric acid in nickel sulfamate plating solutions. Methods are given here that provide acceptable analysis and monitoring of these chemical species in this nickel sulfamate plating process. The optimum respective operating range of nickel, nickel chloride hexahydrate and boric acid are 53.0 - 94.0 g/l, 6.0 - 30.0 g/l, and 30.0 - 45.0 g/l. The resulting precisions are in the range of 0 - 0.5 g/l providing adequate monitoring of these plating solutions supported by two years of testing.

Keywords

chemical analysis, nickel, nickel chloride hexahydrate, boric acid, nickel sulfamate plating solutions, titration

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Introduction

The chemical literature lacks analysis methods for the determination and adequate monitoring of nickel, nickel chloride hexahydrate and boric acid in nickel sulfamate plating solutions. Lack of optimization of these plating solutions causes serious problems for the nickel plating industry such as poor quality products and wasted resources.

The manufacturer of these nickel sulfamate plating solution chemicals uses a quick qualitative test method for the determination of nickel, nickel chloride hexahydrate and boric acid in these nickel sulfamate plating solutions (ref 1). This qualitative test method is not an adequate method.

Titration methods are given here that provide acceptable analysis and monitoring of these chemical species in this nickel sulfamate plating process. General theory and background on titration is extensive (ref 2-5).

Approach

Strict analytical chemistry methods and procedures are followed throughout this experimental section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 2).

Three analytical reagent grade standard solutions are required. They are 0.100 M disodium EDTA, 0.100 N silver nitrate, and 0.100 N sodium hydroxide solutions. Many references are available for information related to preparation and standardization of these reagent grade standard solutions (ref 2-7).

Seven other reagent grade materials are also required. They are ammonium buffer solution consisting of 1 M ammonium sulfamate and 1 M ammonium hydroxide, Murexide indicator tablets consisting of 0.4 mg Murexide combined with potassium sulfamate, dichlorofluorescein solution consisting of a two percent ethanol solution of dichlorofluorescein, BTB/BCP mixed indicator solution consisting of 0.2 percent bromthymol blue and 1.0 percent bromcresol purple in ethanol, solutions of 0.1 N sodium hydroxide and 0.1 N hydrochloric acid, and powered mannitol.

There are separate analytical procedures for each of nickel, nickel chloride hexahydrate, and boric acid in nickel sulfamate plating solutions.

The analytical procedure for the determination of nickel in sample nickel sulfamate plating solutions requires that 1 ml of sample solution is micropipetted into a 250 ml erlenmeyer flask. To this flask, add 50 ml of distilled water, a stirring bar, 10 ml of

ammonium buffer solution, and one Murexide indicator tablet. Dissolve flask contents by swirling. Titrate with 0.100 M disodium EDTA reagent grade standard solution from a yellowish-green to a deep purple endpoint and record the amount of titrant dispensed.

The analytical procedure for the determination of nickel chloride hexahydrate in sample nickel sulfamate plating solutions requires that 20 ml of sample solution is pipetted into a 250 ml erlenmeyer flask. To this flask, add a stirring bar and 1 ml of dichlorofluorescein solution. Titrate with 0.100 N silver nitrate reagent grade standard solution to a pinkish tan endpoint ignoring the pink color of the precipitate and record the amount of titrant dispensed.

The analytical procedure for the determination of boric acid in sample nickel sulfamate plating solutions requires that 2 ml of sample solution is micropipetted into a 250 ml erlenmeyer flask. To this flask, add a stirring bar and 5 drops of BTB/BCP mixed indicator solution. The pH of the mixture in the flask must be 4.1 - 4.3 and can be adjusted with either the 0.1 N sodium hydroxide or 0.1 N hydrochloric acid solutions. Also add to this flask, 5 grams of powered mannitol and mix flask contents until a thick paste is formed. Titrate with 0.100 N sodium hydroxide to a purple endpoint and record the amount of titrant dispensed.

All sample solutions are analyzed in triplicate. Nickel, nickel chloride hexahydrate, and boric acid concentrations in the samples are calculated by their normal chemical stoichiometry.

Results and Discussion

Experimental titration data is presented in Tables 1-3 for the determination of nickel, nickel chloride hexahydrate, and boric acid in a sample nickel sulfamate plating solution, respectively.

In Table 1, the calculation for the nickel concentration in the nickel sulfamate plating solution is:

$$\text{g/l Ni} = (\text{ml 0.1 M } Na_2EDTA \text{ titrant})(5.85) \qquad (1)$$

From equation 1, the value of 76.73 g/l nickel is calculated for the sample solution from the data given in Table 1.

In Table 2, the calculation for the nickel chloride hexahydrate concentration in the nickel sulfamate plating solution is:

$$\text{g/l } NiCl_2\text{-}6H_2O = (\text{ml 0.1 N } AgNO_3 \text{ titrant})(0.60) \qquad (2)$$

From equation 2, the value of 17.89 g/l nickel chloride hexahydrate is calculated for the sample solution from the data given in Table 2.

In Table 3, the calculation for the boric acid concentration in the nickel sulfamate plating solution is:

$$\text{g/l Boric Acid} = (\text{ml 0.1 N NaOH titrant})(3.08) \quad (3)$$

From equation 3, the value of 37.68 g/l boric acid is calculated for the sample solution from the data given in Table 3.

It is useful to evaluate the variations in precision for the materials and methods used. Tables 4-10 present these variations for the 1 ml micropipette, 20 ml class A pipette, 2 ml micropipette, 50 ml class A burette, 0.100 M disodium EDTA standard solution, 0.100 N silver nitrate standard solution, and the 0.100 N sodium hydroxide standard solution, respectively.

The data shows that these methods are sufficient to adequately monitor the nickel, nickel chloride hexahydrate, and boric acid in these nickel sulfamate plating processes. The optimum respective operating range of nickel, nickel chloride hexahydrate, and boric acid are 53.0 - 94.0 g/l, 6.0 - 30.0 g/l, and 30.0 - 45.0 g/l. The resulting precisions are in the range of 0 0- 0.5 g/l providing adequate monitoring of these plating solutions.

Table 1. Experimental Titration Data for Nickel in Nickel Sulfamate Plating Solutions

Replicate	0.100 M Na_2EDTA Titrant Used (ml)	Nickel Conc (g/l)
1	13.05	76.34
2	13.15	76.93
3	13.15	76.93
X(avg)	13.12	76.73
Sn	0.05	0.28

Table 2. Experimental Titration Data for Nickel Chloride Hexahydrate in Nickel Sulfamate Plating Solutions

Replicate	0.100 N $AgNO_3$ Titrant Used (ml)	Nickel Chloride Hexahydrate Conc (g/l)
1	29.85	17.91
2	29.80	17.88
3	29.80	17.88
X(avg)	29.82	17.89
Sn	0.02	0.01

Table 3. Experimental Titration Data for Boric Acid in Nickel Sulfamate Plating Solutions

Replicate	0.100 N NaOH Titrant Used (ml)	Boric Acid Conc (g/l)
1	12.25	37.73
2	12.20	37.58
3	12.25	37.73
X(avg)	12.23	37.68
Sn	0.02	0.07

Table 4. Precision of Micropipetting One Milliliter

Replicate	Volume (ml)
1	1.015
2	1.010
3	1.010
4	1.005
5	1.005
6	1.005
X(avg)	1.008
Sn	0.004

Table 5. Precision of Pipetting Twenty Milliliters

Replicate	Volume (ml)
1	20.00
2	20.05
3	20.05
4	20.00
5	20.05
6	20.05
X(avg)	20.03
Sn	0.02

Table 6. Precision of Micropipetting Two Milliliters

Replicate	Volume (ml)
1	1.995
2	2.005
3	2.005
4	2.005
5	2.000
6	2.000
X(avg)	2.002
Sn	0.004

Table 7. Precision of a 50 ml Class-A Burette

Replicate	Volume (ml)
1	24.94
2	24.98
3	25.02
4	25.05
5	24.98
6	25.05
X(avg)	25.00
Sn	0.04

Volumes are calculated from the weight-volume relationship of a contained deionized water solution corrected for temperature.

Table 8. Precision of 0.100 M Disodium EDTA Standard Solution by Titration

Replicate	Na_2EDTA Conc.(M)*
1	0.105
2	0.100
3	0.100
4	0.100
5	0.105
6	0.100
X(avg)	0.102
Sn	0.002

Table 9. Precision of 0.100 N Silver Nitrate Standard Solution by Titration

Replicate	$AgNO_3$ Conc.(N)*
1	0.100
2	0.100
3	0.100
4	0.105
5	0.105
6	0.100
X(avg)	0.102
Sn	0.002

Table 10. Precision of 0.100 N Sodium Hydroxide Standard Solution by Titration

Replicate	NaOH Conc.(N)*
1	0.105
2	0.100
3	0.100
4	0.100
5	0.100
6	0.100
X(avg)	0.101
Sn	0.002

* Concentrations are calculated using a standard chemical analysis method from Peters (3).

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